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**RECRYSTALLIZATION AND GRAIN-  
GROWTH BEHAVIOR OF A  
NICKEL-BASE SUPERALLOY  
DURING MULTI-HIT DEFORMATION  
(Preprint)**



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# Recrystallization and Grain-Growth Behavior of a Nickel-Base Superalloy During Multi-Hit Deformation

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## Abstract

The initial breakdown behavior of Waspaloy–ingot material with coarse, columnar grain structure was established via isothermal hot compression of double-cone samples. Temperature (1177 °C), strain rate ( $0.1 \text{ s}^{-1}$ ), dwell time between increments of deformation (30 or 60 seconds), and forging direction relative to the columnar structure were typical of industrial practice. Recrystallization kinetics were more rapid during multi-hit testing than monotonic testing. The recrystallization and grain-growth behavior showed a complex dependence on imposed strain, test orientation, and dwell time.

*Keywords:* nickel, recrystallization, particle stimulated nucleation, grain growth, high temperature deformation

## Introduction

The ability to predict and control microstructure during the thermomechanical processing of superalloys is critical to the development of attractive service properties. For example, prior work on both cast and wrought alloy 718 and wrought Waspaloy [1-4] has sought to quantify recrystallization and grain-growth behavior including the effects of multi-step deformation and final annealing on microstructure evolution. Recent efforts on Waspaloy *ingot* material utilizing *monotonic* deformation have provided a fundamental understanding of the effect of texture on recrystallization and flow behavior during deformation [5]. However, coarse-grained, cast ingot may go through numerous operations comprising upsetting, drawing-out, and reheating in order to achieve a uniformly recrystallized microstructure. Additional work to establish the relationship between industrial processing parameters and microstructure evolution is therefore needed. Hence, the present work was undertaken to meet the need for a typical superalloy ingot material. Specifically, isothermal, *multi-hit*, hot compression tests on coarse-grain Waspaloy-ingot material were conducted to provide insight into the effects of process parameters more representative of industrial practice than monotonic deformation on microstructure evolution.

## Material and Procedures

The material used for this program was from the same 508-mm-diameter, 2500-mm-long Waspaloy ingot synthesized via vacuum induction melting followed by vacuum arc remelting (VIM/VAR) and homogenization treatment as used in a previous effort [5]. The measured composition (in wt. pct.) was 1.40 aluminum, 2.94 titanium, 19.5



chromium, 13.3 cobalt, 3.98 molybdenum, 0.49 iron, 0.037 carbon, 0.0046 nitrogen, 0.0002 oxygen, balance nickel. The program material had a columnar-grain microstructure with a solidification texture typical of fcc alloys [6]. The cast and homogenized grain size was  $4.8 \pm 2.6$  mm diameter  $\times$   $43.6 \pm 17.5$  mm length. The material also contained a dispersion of carbide particles comprising  $\sim 0.25$  volume pct.

Double-cone hot compression tests were conducted to establish the effect of industrial-type processing conditions on microstructure evolution. This simulative test was chosen because it provides a wide distribution of strain across the sample, and thus the required number of samples was minimal compared to characterization via conventional cylindrical upset tests. The double-cone samples (Figure 1) were machined from various regions of the ingot such that the columnar-grain orientations lay at 0, 90, or 45° with respect to the compression axis. Designated axial, transverse, and 45°, respectively, these orientations are typical of those pertinent to ingot breakdown operations (Figure 2).

All of the hot compression experiments were conducted using a 900 kN servohydraulic test frame with 83-mm-diameter TZM-molybdenum dies. The sample and dies were induction heated with a susceptor in a nitrogen atmosphere. The die stack was coated with magnesia to prevent oxidation; boron nitride was used as a lubricant and parting agent for the test samples. Prior to compression, the die stack and sample were heated and allowed to soak for 45 minutes at test temperature.

Multi-hit testing was conducted at a temperature (1177°C) and strain rate ( $0.1 \text{ s}^{-1}$ ) typical of industrial practice. Strain increments of 0.15 relative to the original double-cone height,  $h_0$ , and dwell times between hits of 30 or 60 seconds were employed.

Each sample was incrementally upset to a 3:1 total reduction. After the final strain increment, the sample was water-quenched within 2 to 3 seconds.

Following testing, each sample was sectioned parallel to the compression axis and prepared using standard metallographic techniques for optical and scanning-electron microscopy (SEM). Because of the tendency for the samples to develop a small degree of ovality during testing [5], sections were taken along two orthogonal directions parallel to the principal axes of ovality. Optical micrographs at 25X were shot on polished-and-etched sections at the mid-height for a series of locations across the diameter of the sample. The fraction recrystallized and the grain size were determined from these micrographs using FoveaPro [7]; these measurements were also corroborated via electron-backscatter diffraction (EBSD) in an SEM. For this purpose, a Leica Stereoscan 360 field-emission gun (FEG) SEM [8], equipped with an electron back scatter diffraction (EBSD) detector and TSL-OIM version 3.5 software [9], was used.

In order to correlate the observed microstructures to local deformation conditions, finite-element-method (FEM) simulations of the double-cone test were conducted. Specifically, DEFORM 2D [10] simulations were used to determine the local von Mises effective strain profile along the midplane. All simulations assumed isotropic material conditions. To quantify the effect of ovaling on predicted strain values, additional, more-detailed, FEM simulations utilizing an approximate anisotropic yield function and flow rule were conducted. Comparison of the isotropic and anisotropic FEM simulations revealed that the simpler isotropic approach provided a reasonable engineering estimate of local strains and thus was used to interpret the results reported below.



## Results and Discussion

The principal results obtained from the multi-hit testing consisted of observations of recrystallization kinetics and final grain size.

**Recrystallization Kinetics.** Optical metallography on the double-cone samples revealed a variation of fraction recrystallized along the sample diameter and hence a noticeable dependence on strain. At low strains ( $\sim 0.3$ - $0.4$ ), the recrystallization appeared to have originated as a result of particle-stimulated nucleation (PSN) at carbides. The specific strain for nucleation was independent of the test-sample direction relative to the columnar grain orientation to a first order. This finding was surprising in view of previous observations of an orientation dependence of the overall dynamic recrystallization kinetics for monotonic tests [5].

The fraction recrystallized in the multi-hit tests showed an Avrami-type (sigmoidal) dependence on strain (Figure 3). Despite the unavoidable scatter associated with quantifying the recrystallization behavior of textured, coarse-grain materials, there was a measurable dependence of fraction recrystallized on orientation, unlike the PSN observations. Specifically, the rate of recrystallization was slowest for the axial samples and fastest for the  $45^\circ$  samples for a dwell time of 30 seconds (Figure 3a). On the other hand, the trend was less apparent for the dwell time of 60 seconds (Figure 3b). This difference may be due to the limited sampling statistics with coarse-grain material. In comparison to the previous monotonic observations [5], however, the magnitude of the dependence on orientation was significantly *less*. This reduction in orientation dependence for the multi-hit versus the monotonic tests was mirrored by the

strains for 50 pct recrystallization (Table 1). Table 1 also reveals a small (second-order) effect of dwell time on the strain for 50 pct. recrystallization. Such a finding suggests that either metadynamic recrystallization controls the behavior between the deformation steps, or that classical static recrystallization occurs during intervals of time substantially less than or equal to the shortest dwell time, i.e. 30 seconds. These findings are consistent with previous measurements of metadynamic/static recrystallization for wrought Waspaloy [4].

Despite the observed sigmoidal dependence of fraction recrystallized on strain for multi-hit deformation, it is difficult to draw a conclusion regarding the specific mechanisms controlling microstructure evolution in such thermomechanical processes. It may be hypothesized that dynamic, metadynamic, and static recrystallization are the important mechanisms controlling microstructure evolution, albeit coupled in a complex manner. Nevertheless, the relative importance of dynamic versus static recrystallization may be estimated via comparison with previous results for strictly dynamic recrystallization of Waspaloy-ingot material [5]. For example, the observed kinetics for both multi-hit and monotonic deformation of axial samples at 1177 °C,  $0.1\text{s}^{-1}$ , are compared in Figure 4. The increase in rate was almost a factor of 2, a trend which can be ascribed to the additional influence of metadynamic and static recrystallization during multi-hit deformation. On the other hand, the increment in recrystallization rate was a factor of ~1.5 or ~1.6 for transverse and 45° samples, respectively. The reduced sensitivity of the kinetics of recrystallization during monotonic versus multi-hit deformation for these two orientations suggests that DRX is dominant relative to both MDRX and SRX. This trend, however, is not unexpected due to the rapid kinetics



observed during monotonic testing of samples with these orientations compared to the relatively slower DRX kinetics observed for axial samples.

The observed kinetics for multi-hit deformation of coarse-grain Waspaloy ingot material were also compared to the dynamic recrystallization kinetics for fine-grain, wrought Waspaloy [4] (Figure 5). This comparison shows almost identical behavior for the wrought Waspaloy and multi-hit, coarse-grain 45° samples. The recrystallization kinetics for the axial and transverse ingot samples were also relatively close to the kinetics for the wrought material.

The behavior shown in Figure 5 can be interpreted in terms of the various nucleation and recrystallization events that control microstructure evolution for the cast-versus-wrought materials. First, observations from Reference 4 indicate that nucleation of dynamic recrystallization occurs at smaller strains in the wrought material. This fact alone suggests that the overall DRX plot for the coarse-grain material would lie to the right (i.e., at higher strains) compared to the wrought material. On the other hand, recent cellular-automaton simulations of DRX have indicated that the Avrami exponents of fine-grain wrought material are comparable to those of coarse-grain ingot material (with 0.25 volume pct carbides) as a result of PSN in the latter material [11]. Hence, in the absence of static recrystallization, the curves for the wrought and ingot material in Figure 5 would be expected to be parallel but displaced by an amount at least equal to the difference in strain for initial nucleation. However, the fact that the slopes are steeper for the multi-hit cast material may indicate an enhancement of overall recrystallization rate as a result of static recrystallization between increments of deformation.

**Recrystallized Grain Size.** The recrystallized structures from the multi-hit tests revealed measurable, but not large, differences in grain size compared to previous observations for monotonic tests [5] (Table 2). Not surprisingly, the dynamically recrystallized grain sizes from the monotonic tests were *comparable* to those from the multi-hit tests, i.e., both were  $\sim 85\ \mu\text{m}$ . On the other hand, the multi-hit grain sizes were *finer* than those observed for the monotonic tests with post-deformation soak times of 30 seconds ( $\sim 160\ \mu\text{m}$ ) or 5 minutes ( $\sim 430\ \mu\text{m}$ ). This comparison can be rationalized on the basis of the fact the monotonic-test grain structures at 30 seconds, let alone 5 minutes, have evolved as a result of metadynamic/static recrystallization *and* grain growth [4]. By contrast, the multi-hit tests incorporated 30 or 60 second dwell times and water quenching *immediately* following the final increment of deformation. The fact that the recrystallized grain sizes in the multi-hit tests were smaller than those developed during monotonic testing with a 30 second soak therefore suggests that dynamic (and perhaps metadynamic) recrystallization play a more important role compared to grain growth in determining grain size developed once the final increment of deformation has been imposed. Further experimental and theoretical work is now underway to distinguish the effect of metadynamic/static recrystallization from grain growth on microstructure evolution at short times.

### Summary and Conclusions

Incremental reduction, isothermal double-cone compression tests were performed at supersolvus temperatures to establish recrystallization behavior as a function of orientation relative to the initial columnar microstructure in a cast-and-



homogenized ingot of a typical gamma-gamma prime superalloy, Waspaloy. The following conclusions were drawn from this work.

1. As for monotonic deformation, the recrystallization kinetics were dependent on test direction, but to a lesser degree. For multi-hit tests involving a 30-second dwell time, axially-oriented samples were slowest to recrystallize, and 45° samples fastest, as was observed previously for monotonic deformation. A similar trend was found for low strain regions in 60-second dwell time samples. For higher strains, however, the transverse sample orientation recrystallized fastest, while the axial samples still recrystallized the slowest.
2. For a given test orientation, multi-hit recrystallization kinetics are more rapid than monotonic kinetics because of the beneficial influence of MDRX and SRX. The specific effect of dwell time per se on recrystallization during multi-hit deformation is limited because of the large influence of MDRX.
3. Multi-hit recrystallization kinetics for coarse-grained ingot material are comparable to those for DRX of wrought material because of the beneficial influence of MDRX and PSN.
4. In samples quenched immediately after deformation, the recrystallized grain size is essentially identical for monotonic *and* multi-hit tests for a given strain rate and temperature.

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## References

1. M.J. Weis, M.C. Mataya, S.W. Thompson, and D.K. Matlock: *Superalloy 718 – Metallurgy and Applications*, ed. by E.A. Loria, TMS, Warrendale, PA, 1989, pp. 135-54.
2. M.C. Mataya: *Superalloy 718 – Metallurgy and Applications*, ed. by E.A. Loria, TMS, Warrendale, PA, 1989, 155-78.
3. D. Zhao, S. Guillard, and A.T. Male: *Superalloys 718, 625, 706 and Various Derivatives*, ed. by E.A. Loria, TMS, Warrendale, PA, 1997, pp. 193-204.
4. G. Shen, S.L. Semiatin, and R. Shivpuri: *Metall. Trans. A*, 1995, vol. 26A, pp. 1795-803.
5. S.L. Semiatin, et.al: *Metall. Trans. A*, 2004, vol. 35A, pp. 679-93.
6. M.G. Glavicic, P.A. Kobryn, F. Spadafora, and S.L. Semiatin: *Mater. Sci. Eng. A*, 2003, vol. A346, pp. 8-18.
7. FoveaPro™, Reindeer Graphics, Asheville, NC
8. Cambridge Instruments, Cambridge, UK
9. TexSEM Laboratories, Inc., Provo, Utah
10. DEFORM™ 2D, Scientific Forming Technologies Corporation, Columbus, OH, 1994.
11. R.L. Goetz: *Scripta Mater.*, 2005, vol. 52, pp. 851-856.

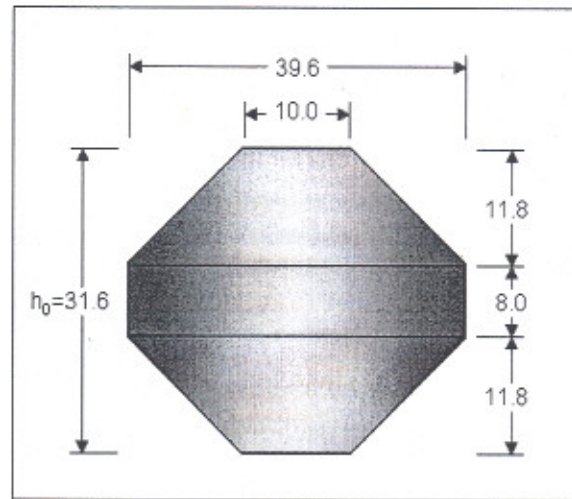


Figure 1. Geometry of the double-cone samples used in the current work. (All dimensions are in millimeters.)



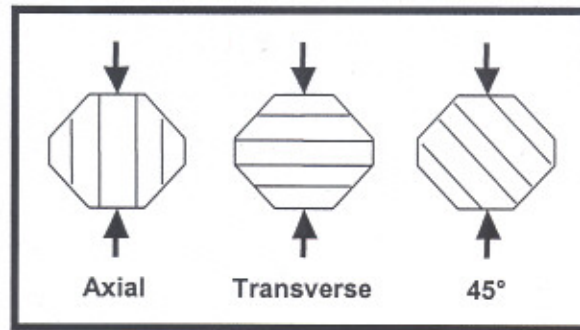
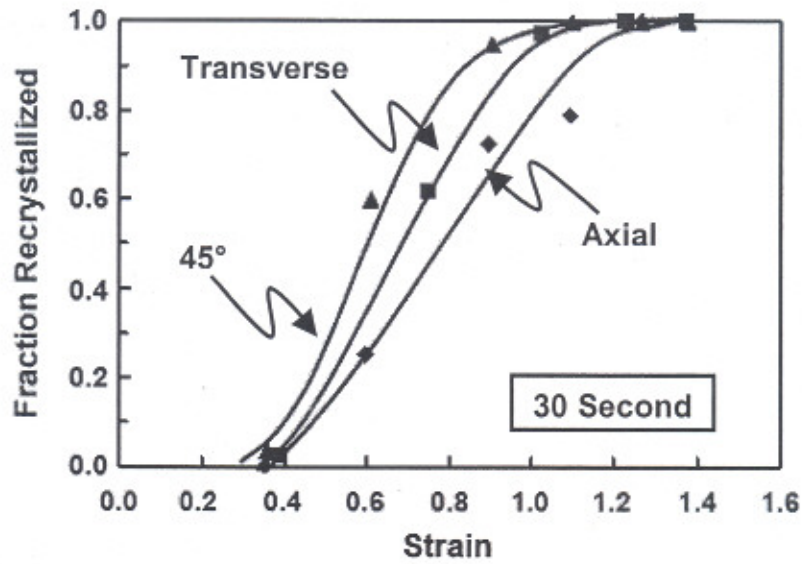
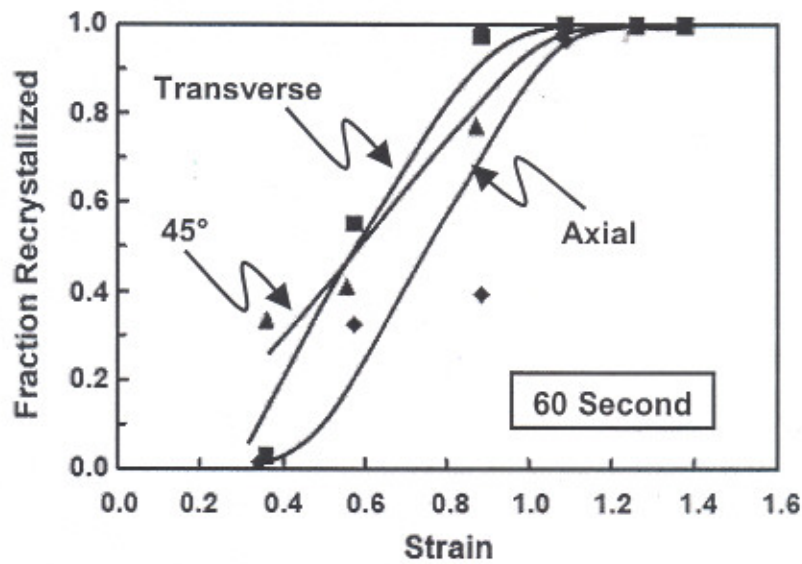


Figure 2. Schematic showing grain orientation-compression axis relationships during testing and sample-orientation nomenclature. Arrows indicate compression direction.



(a)



(b)

Figure 3. Dependence of fraction recrystallized on strain and test orientation of Waspaloy ingot material deformed at 1177°C,  $0.1\text{s}^{-1}$ , with either (a) 30 second or (b) 60 second dwell time at temperature between deformation increments.

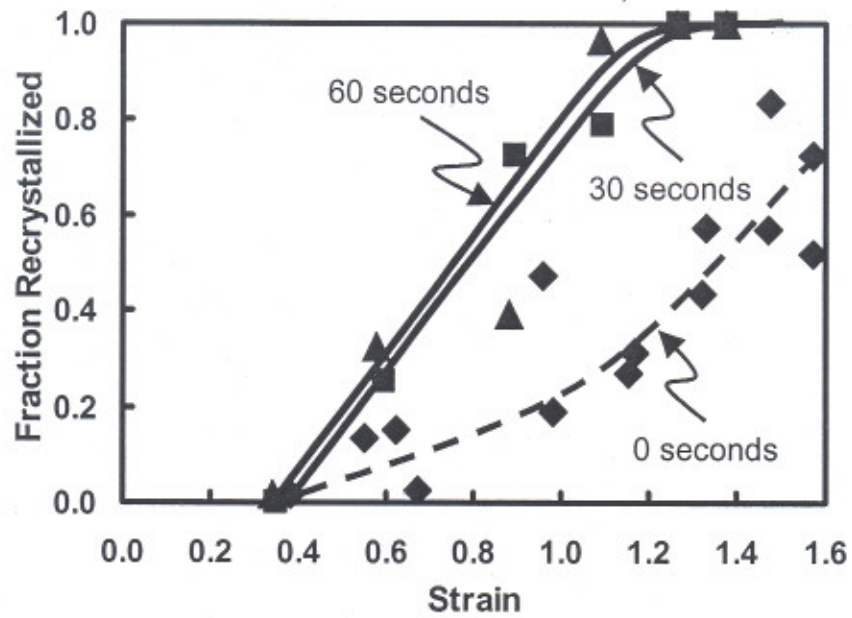


Figure 4. Dependence of fraction recrystallized on strain and dwell time between deformation increments of *axially*-oriented Waspaloy ingot material deformed at 1177°C, 0.1 s<sup>-1</sup>. The results for the corresponding axially-oriented monotonic test (broken line) are compared to those for the multi-hit tests (solid lines).



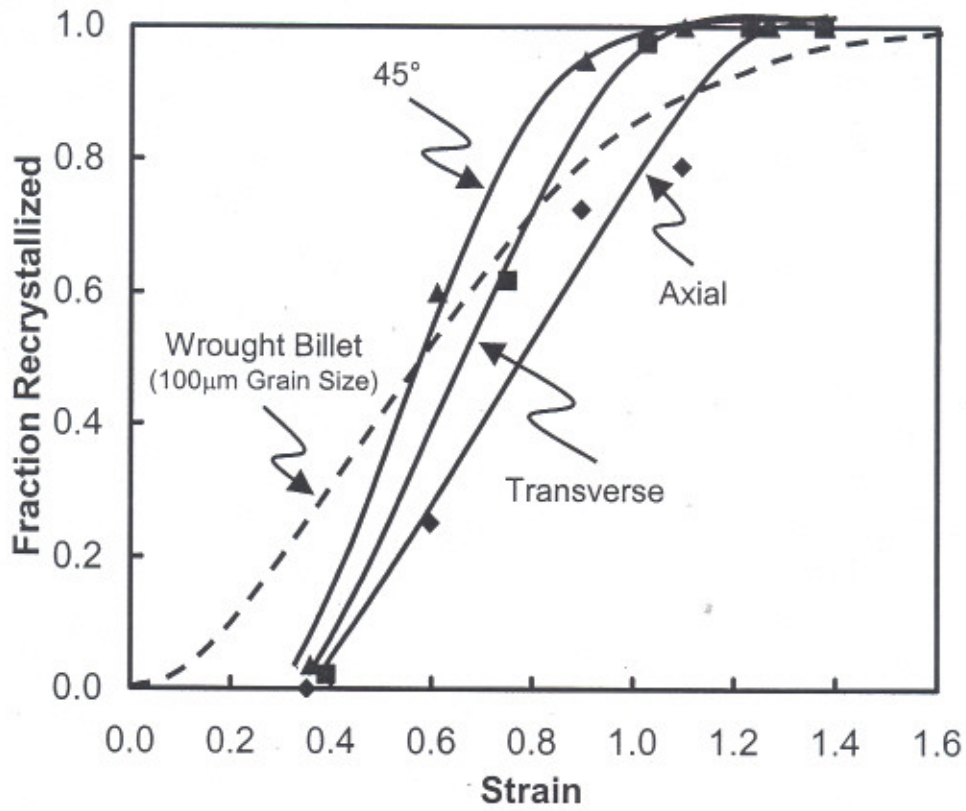


Figure 5. Dependence of fraction recrystallized on strain and test orientation in Waspaloy ingot material deformed at  $1177^{\circ}\text{C}$  and  $0.1\text{s}^{-1}$  using the multi-hit approach with 30 second dwell time. The multi-hit results are compared to those for monotonically-deformed wrought Waspaloy with a  $100\text{-}\mu\text{m}$  grain size (broken line).

Table 1. Orientation dependence of strain for 50 percent recrystallized microstructure in Waspaloy ingot material deformed at 1177°C and a strain rate of  $0.1\text{s}^{-1}$

	Dwell Time (s)	Orientation	Strain for 50% RX
<b>Monotonic</b>	0	Wrought*	0.55
	0	Axial	1.35
	0	Transverse	0.9
	0	45°	0.75
<b>Multi-Hit</b>	30	Axial	0.8
	30	Transverse	0.7
	30	45°	0.6
	60	Axial	0.75
	60	Transverse	0.55
	60	45°	0.6

\*100- $\mu\text{m}$  starting grain size

Table 2. Dependence of recrystallized grain size on dwell time and orientation for Waspaloy ingot material deformed at 1177°C and a strain rate of 0.1s<sup>-1</sup>

	Dwell Time (seconds)*	Axial (μm)	Transverse (μm)	45° (μm)	Average (μm)
<b>Monotonic</b>	0	90	90	80	87
	30	150	190	145	162
	300	395	425	475	430
<b>Multi-Hit</b>	30	90	75	80	82
	60	80	100	80	87

\* Dwell time is the time between blows in the multi-hit tests and the final dwell time at the end of monotonic tests prior to water quenching.